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[272] Attorney Docket No. : ADP-238 PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant	:	Donald J. Ames, Ellen K. Brackman, and Donald L. Guile
Serial No.	:	10/738,425
Filed	:	December 16, 2003
For	:	CREEP RESISTANT ZIRCON REFRACTORY MATERIAL USED IN A GLASS MANUFACTURING SYSTEM
Examiner	:	David R. Sample
Group	:	1755

SUPPLEMENTAL INFORMATION DISCLOSURE STATEMENT

Submitted herewith is a Petition Under 37 CFR §1.313(c) and the required fee requesting that this application be withdrawn from issuance for consideration of the present Supplemental Information Disclosure Statement.

This Information Disclosure Statement relates to commonly-assigned U.S. patent application number 11/294,315 (the '315 application). Attached hereto as Exhibit A is a copy of a February 8, 2006 Office Action regarding the '315 application. In paragraph 3 of that Office Action, the examiner in charge of the '315 application entered an obviousness-type double patenting rejection based on the present application. The applicants for the '315 application believe that this obviousness-type double patenting should be withdrawn and have today submitted a response to the February 8th Office Action setting forth the reasons why they believe the rejection is in error. A copy of that submission is attached as Exhibit B (see, in particular, pages 4-5).

In the February 8th Office Action for the '315 application, the examiner also made an obviousness-type double patenting rejection based on U.S. Patent No. 6,974,786 (the '786 patent), and a terminal disclaimer for the '315 application directed to the '786 patent was submitted today along with the amendment of Exhibit B. The '786 patent claims priority from PCT/US01/45300 which was previously made of record in the present application (see WO 02/44102 which was submitted at the time this

application was filed, i.e., on December 16, 2003). For the examiner's convenience, a copy of the '786 patent is submitted herewith as Exhibit C.

Consideration of the foregoing in connection with the prosecution of this application is respectfully requested.

Respectfully submitted,

Date: 5/8/06

Maurice Klee

Maurice M. Klee, Ph.D.
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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
11/294,315	12/05/2005	John D. Helfinstine	ADP-165.1US	6216

23520 7590 02/08/2006

MAURICE M KLEE
1951 BURR STREET
FAIRFIELD, CT 06824

EXAMINER	
GROUP, KARL E	
ART UNIT	**PAPER NUMBER**
1755	

DATE MAILED: 02/08/2006

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Please find below and/or attached an Office communication concerning this application or proceeding.

PTO-90C (Rev. 10/03)

Exhibit A

Office Action Summary	Application No.	Applicant(s)	
	11/294,315	HELFINSTINE ET AL.	
	Examiner	Art Unit	
	Karl E. Group	1755	

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

Period for Reply

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) OR THIRTY (30) DAYS, WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

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Status

1)☐ Responsive to communication(s) filed on _____.

2a)☐ This action is FINAL. 2b)☒ This action is non-final.

3)☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

Disposition of Claims

4)☒ Claim(s) 1-23 is/are pending in the application.

4a) Of the above claim(s) _____ is/are withdrawn from consideration.

5)☐ Claim(s) _____ is/are allowed.

6)☒ Claim(s) 1-23 is/are rejected.

7)☐ Claim(s) _____ is/are objected to.

8)☐ Claim(s) _____ are subject to restriction and/or election requirement.

Application Papers

9)☐ The specification is objected to by the Examiner.

10)☐ The drawing(s) filed on _____ is/are: a)☐ accepted or b)☐ objected to by the Examiner.

Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).

Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).

11)☐ The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

Priority under 35 U.S.C. § 119

12)☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).

a)☐ All b)☐ Some * c)☐ None of:

1.☐ Certified copies of the priority documents have been received.

2.☐ Certified copies of the priority documents have been received in Application No. _____.

3.☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

* See the attached detailed Office action for a list of the certified copies not received.

Attachment(s)

1) ☒ Notice of References Cited (PTO-892)
2) ☐ Notice of Draftsperson's Patent Drawing Review (PTO-948)
3) ☒ Information Disclosure Statement(s) (PTO-1449 or PTO/SB/08) Paper No(s)/Mail Date 12-5-05.
4) ☐ Interview Summary (PTO-413) Paper No(s)/Mail Date. _____.
5) ☐ Notice of Informal Patent Application (PTO-152)
6) ☐ Other: _____.

Specification

1. The disclosure is objected to because of the following informalities: Applicants are requested to amend the disclosure to update the status of the parent application.

Appropriate correction is required.

Double Patenting

2. The nonstatutory double patenting rejection is based on a judicially created doctrine grounded in public policy (a policy reflected in the statute) so as to prevent the unjustified or improper timewise extension of the "right to exclude" granted by a patent and to prevent possible harassment by multiple assignees. A nonstatutory obviousness-type double patenting rejection is appropriate where the conflicting claims are not identical, but at least one examined application claim is not patentably distinct from the reference claim(s) because the examined application claim is either anticipated by, or would have been obvious over, the reference claim(s). See, e.g., In re Berg, 140 F.3d 1428, 46 USPQ2d 1226 (Fed. Cir. 1998); In re Goodman, 11 F.3d 1046, 29 USPQ2d 2010 (Fed. Cir. 1993); In re Longi, 759 F.2d 887, 225 USPQ 645 (Fed. Cir. 1985); In re Van Ornum, 686 F.2d 937, 214 USPQ 761 (CCPA 1982); In re Vogel, 422 F.2d 438, 164 USPQ 619 (CCPA 1970); and In re Thorington, 418 F.2d 528, 163 USPQ 644 (CCPA 1969).

A timely filed terminal disclaimer in compliance with 37 CFR 1.321(c) or 1.321(d) may be used to overcome an actual or provisional rejection based on a nonstatutory double patenting ground provided the conflicting application or patent either is shown to be commonly owned with this application, or claims an invention made as a result of activities undertaken within the scope of a joint research agreement.

Effective January 1, 1994, a registered attorney or agent of record may sign a terminal disclaimer. A terminal disclaimer signed by the assignee must fully comply with 37 CFR 3.73(b).

3. Claims 1-23 are provisionally rejected on the ground of nonstatutory obviousness-type double patenting as being unpatentable over claims 1-28 of copending Application No. 10/738425. Although the conflicting claims are not identical, they are not patentably distinct from each other because the scope of the copending claims overlap the instant claims, note the content of TiO_2. Furthermore, the instant claims use the terminology "comprising" therefor not excluding the iron oxide.

This is a provisional obviousness-type double patenting rejection because the conflicting claims have not in fact been patented.

4. Claims 1-3,6,7,17-19,22-23 are rejected on the ground of nonstatutory obviousness-type double patenting as being unpatentable over claims 1-23 of U.S. Patent No. 6,974,786. Although the conflicting claims are not identical, they are not patentably distinct from each other because the scope of the instant claims and the patented claims overlap.

The copending and patented claims and the claims differ in that they do not teach the exact same proportions as recited in the instant claims.

However, one of ordinary skill in the art at the time the invention was made would have considered the invention to have been obvious because the compositional proportions taught by copending and patented claims overlap the instantly claimed proportions and therefore are considered to establish a prima facie case of obviousness. It would have been obvious to one of ordinary skill in the art to select any portion of the disclosed ranges including the instantly claimed ranges from the ranges disclosed in the prior art reference, particularly in view of the fact that;

> "The normal desire of scientists or artisans to improve upon what is already generally known provides the motivation to determine where in a disclosed set of percentage ranges is the optimum combination of percentages", In re Peterson 65 USPQ2d 1379 (CAFC 2003).

Also, In re Geisler 43 USPQ2d 1365 (Fed. Cir. 1997); In re Woodruff, 16 USPQ2d 1934 (CCPA 1976); In re Malagari, 182 USPQ 549, 553 (CCPA 1974) and MPEP 2144.05.

5. A rejection based on double patenting of the "same invention" type finds its support in the language of 35 U.S.C. 101 which states that "whoever invents or

discovers any new and useful process ... may obtain a patent therefor ..." (Emphasis added). Thus, the term "same invention," in this context, means an invention drawn to identical subject matter. See *Miller v. Eagle Mfg. Co.*, 151 U.S. 186 (1894); *In re Ockert*, 245 F.2d 467, 114 USPQ 330 (CCPA 1957); and *In re Vogel*, 422 F.2d 438, 164 USPQ 619 (CCPA 1970).

A statutory type (35 U.S.C. 101) double patenting rejection can be overcome by canceling or amending the conflicting claims so they are no longer coextensive in scope. The filing of a terminal disclaimer cannot overcome a double patenting rejection based upon 35 U.S.C. 101.

6. Claims 4,5,8-16,20,21 are rejected under 35 U.S.C. 101 as claiming the same invention as that of claims 4,5,8-16,20,21of prior U.S. Patent No. 6,974,786. This is a double patenting rejection. The rejected claims have the same scope of the patented claims. Dependent claims 4,5,20 and 21 require the same creep rate as the corresponding patented claims.

7. Any inquiry concerning this communication or earlier communications from the examiner should be directed to Karl E. Group whose telephone number is 571-272-1368. The examiner can normally be reached on M-F (6:30-4:00) First Friday Off.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Jerry Lorengo can be reached on 571-272-1233. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see http://pair-direct.uspto.gov. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free).

Karl E Group
Primary Examiner
Art Unit 1755

Keg
2-6-0

[272] Attorney Docket No. : ADP-165.1US PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant	:	John D. Helfinstine, Daniel J. Liebner, John L. Martin, Dean V. Neubauer, and William R. Powell
Appl. No.	:	11/294,315
Filed	:	December 5, 2005
For	:	SAG CONTROL OF ISOPIPES USED IN MAKING SHEET GLASS BY THE FUSION PROCESS
Examiner	:	K. Group
Group	:	1755

AMENDMENT

This Amendment is submitted in response to the Office Action dated February 8, 2006. Its contents are:

(1) an amendment to the specification -- page 2; and

(2) applicants' Remarks -- page 3.

No extension of time is believed to be necessary for the filing of this Amendment, but if an extension of time is required, applicants request that this be considered a petition therefor. The Director is hereby authorized to charge any fees which may be required for such an extension to Deposit Account No. 11-1158.

Exhibit B

Amendments to the Specification:

On page 1, please replace the paragraph which appears under the heading "CROSS REFERENCE TO RELATED APPLICATIONS" with the following amended paragraph:

This application is a continuation of U.S. Application No. 10/449,701, filed on May 29, 2003, <u>now U.S. Patent No. 6,974,786,</u> the contents of which are incorporated herein by reference, which is a continuation of International Application No. PCT/US01/45300, filed on November 30, 2001, which was published in English under PCT Article 21(2) on June 6, 2002 as International Publication No. WO 02/44102. This application claims the benefit under 35 USC §119(e) of U.S. Provisional Application No. 60/250,921, filed on December 1, 2000.

REMARKS

The undersigned attorney would like to thank Examiner Group for the courteous telephone interview conducted on May 1, 2006. During that interview, it was pointed out that the February 8, 2006 Office Action refers to pending Claims 1-23, while in fact there are only 16 claims in this application. Notwithstanding this discrepancy, the rejections made by the Examiner in the Office Action can be understood and responded to. Accordingly, Examiner Group and the undersigned agreed that a response would be filed based on the actual number of claims pending in the application (i.e., 16).

All of the rejections of the February 8th Office Action are double patenting rejections. Two are non-statutory, obviousness-type double patenting rejections and one is a "same invention" double patenting rejection under 35 U.S.C. §101.

Beginning with the "same invention" rejection, that rejection is based on applicants' U.S. Patent No. 6,974,786 (the '786 patent), from which this application claims priority. The '786 patent contains four independent claims, i.e., Claims 1 , 8, 15, and 17. Independent Claims 1, 8, and 17 each require that the isopipe comprises a zircon refractory which:

> has a mean creep rate at 1180° C. and 250 psi of less than 0.7×10^{-6} inches/inches/hour, where the mean creep rate is determined using a power law model.

None of the claims of this application include this limitation. Accordingly, as to independent Claims 1, 8, and 17, and their dependent claims, there is no "same invention" double patenting.

Instead of the above limitation, independent Claim 15 of the '786 patent calls for a zircon refractory which:

> has a mean creep rate (MCR) at 1180° C. and 250 psi and a 95 percent confidence band (CB) for said mean creep rate such that the CB to MCR ratio is less than 0.5, the MCR and the GB both being determined using a power law model.

Again, none of the claims of the present application include this limitation so none of those claims are claiming the "same invention" as that claimed in the '786 patent.

In view of these considerations, applicants respectfully submit that the "same invention" double patenting rejection based on the '786 patent should be withdrawn.

The February 8th Office Action also included an obviousness-type double patenting rejection based on the '786 patent. To facilitate the prosecution of this application, submitted herewith is a terminal disclaimer for the present application directed to the '786 patent. This terminal disclaimer is being made without prejudice and its sole purpose is to obviate the double patent rejection. Pursuant to the holding in, for example, Quad Environmental Technologies Corp. v. Union Sanitary District, 946 F.2d 870, 20 USPQ2d 1392 (Fed. Cir. 1991), this terminal disclaimer is not to be construed in any way as an obviousness admission with regard to any of the claims of this application or of the '786 patent.

The February 8th Office Action contained a second obviousness-type double patenting rejection, in this case a provisional rejection based on co-pending application number 10/729425 (the '425 application). As noted during the May 1st telephone interview, this double patenting rejection is considered to be peculiar because if the '425 application had had a different assignee, it would not have been cited against the present application in view of its December 16, 2003 filing date which is long after the December 1, 2000 priority date of the present application. Indeed, the subject matter of the present application is prior art to the '425 application and the PCT publication of the present application (i.e., WO 02/44102) was made of record by the applicants for the '425 application during the prosecution of their application.

In her dissent in Eli Lilly & Co. v. Barr Labs., Inc. 251 F.3d 955, 58 USPQ2d 1865, 1866 (Fed. Cir. 2001), Judge Newman discussed the relationship between obviousness-type double patenting and citable art:

> The judgemade law of obviousness-type double patenting was developed to cover the situation where patents are not citable as a reference against each other and therefore can not be examined for compliance with the rule that only one patent is available per invention. Double patenting thus is applied when neither patent is prior art against the other, usually because they have a common priority date. *See General Foods Corp. v. Studiengesellschaft Kohle mbH*, 972 F.2d 1272,

> 1278-81, 23 USPQ2d 1839, 1843-46 (Fed. Cir. 1992) (summarizing the criteria for obviousness-type double patenting). As the court explained in *In re Boylan*, 392 F.2d 1017, 1018 n.1, 157 USPQ 370, 371n.1 (CCPA 1968), "it must always be carefully observed that the appellant's patent is not 'prior art' under either section 102 or section 103 of the 1952 Patent Act." (emphasis added)

Also relevant to the propriety of the Examiner's obviousness-type double patenting rejection based on the '425 application is the MPEP's discussion of the differences between domination and double patenting:

> Domination and double patenting should not be confused. They are two separate issues. One patent or application "dominates" a second patent or application when the first patent or application has a broad or generic claim which fully encompasses or reads on an invention defined in a narrower or more specific claim in another patent or application. Domination by itself, i.e., in the absence of statutory or nonstatutory double patenting grounds, cannot support a double patenting rejection. *In re Kaplan*, 789 F.2d 1574, 1577-78, 229 USPQ 678, 681 (Fed. Cir. 1986); and *In re Sarrett*, 327 F.2d 1005, 1014-15, 140 USPQ 474, 482 (CCPA 1964). However, the presence of domination does not preclude double patenting. See, e.g., *In re Schneller*, 397 F.2d 350, 158 USPQ 210 (CCPA 1968). (MPEP §804)

In light of all these considerations, applicants submit that the obviousness-type double patenting rejection of the present application based on the '425 application is unsound and should be withdrawn. However, if not withdrawn, applicants believe that the rejection should be held in abeyance until the '425 application issues.

In view of the foregoing, reconsideration and the issuance of a notice of allowance for this application are respectfully requested.

Respectfully submitted,

Date: 5/8/06

Maurice Klee

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Reg. No. 30,399
Attorney for Applicant
1951 Burr Street
Fairfield, CT 06824
(203) 255-1400

US006974786B2

(12) **United States Patent**
Helfinstine et al.

(10) **Patent No.:** **US 6,974,786 B2**
(45) **Date of Patent:** **Dec. 13, 2005**

(54) **SAG CONTROL OF ISOPIPES USED IN MAKING SHEET GLASS BY THE FUSION PROCESS**

(75) Inventors: **John D. Helfinstine**, Big Flats, NY (US); **Daniel J. Liebner**, Corning, NY (US); **John L. Martin**, Weston, WV (US); **Dean V. Neubauer**, Horseheads, NY (US); **William R. Powell**, Horseheads, NY (US)

(73) Assignee: **Corning Incorporated**, Corning, NY (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 0 days.

(21) Appl. No.: **10/449,701**

(22) Filed: **May 29, 2003**

(65) **Prior Publication Data**

US 2004/0055338 A1 Mar. 25, 2004

Related U.S. Application Data

(63) Continuation of application No. PCT/US01/45300, filed on Nov. 30, 2001.

(60) Provisional application No. 60/250,921, filed on Dec. 1, 2000.

(51) **Int. Cl.**[7] **C04B 35/78**; C03B 17/06
(52) **U.S. Cl.** **501/106**; 65/374.13; 65/195
(58) **Field of Search** 501/106, 107; 65/374.13, 195

(56) **References Cited**

U.S. PATENT DOCUMENTS

2,746,874 A	5/1956	Whittemore, Jr.	106/57
2,752,259 A	6/1956	Cook	106/57
3,285,757 A	11/1966	Cornely	106/57
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3,347,687 A	10/1967	Henry	106/57
3,359,124 A	12/1967	Henry	106/57
3,437,470 A	4/1969	Overman	65/195
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5,407,873 A	4/1995	Goerenz et al.	501/106
5,466,643 A	11/1995	Ishino et al.	501/105
6,121,177 A *	9/2000	Guigonis et al.	501/105

FOREIGN PATENT DOCUMENTS

JP	11-246230	9/1999

OTHER PUBLICATIONS

Kingery et al., "Plastic Deformation, Viscous Flow, and Creep", *Introduction to Ceramics*, 2[nd] Edition, John Wiley & Sons, New York, 1976, pp. 704-767.

(Continued)

Primary Examiner—Karl Group
(74) *Attorney, Agent, or Firm*—Maurice M. Klee

(57) **ABSTRACT**

Isopipes for use in making sheet glass by a fusion process are provided which exhibit reduced sag. The isopipes are composed of a zircon refractory which has a mean creep rate (MCR) at 1180° C. and 250 psi and a 95 percent confidence band (CB) for said mean creep rate such that the CB to MCR ratio is less than 0.5, the MCR and the CB both being determined using a power law model. The zircon refractory can contain titania (TiO_2) at a concentration greater than 0.2 wt. % and less than 0.4 wt. %. A concentration of titania in this range causes the zircon refractory to exhibit a lower mean creep rate than zircon refractories previously used to make isopipes. In addition, the variation in mean creep rate is also reduced which reduces the chances that the zircon refractory of a particular isopipe will have an abnormally high creep rate and thus exhibit unacceptable sag prematurely.

23 Claims, 5 Drawing Sheets

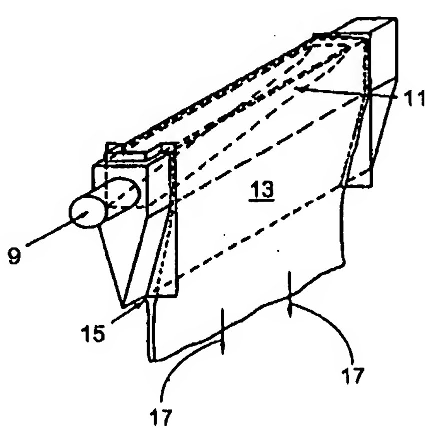

Exhibit C

OTHER PUBLICATIONS

"Flat Glass", *Fundamentals of Inorganic Glasses*, Academic Press, Inc., Boston, 1994, Chapter 20, Section 4.2, pp. 534-540.

Draper et al., *Applied Regression Analysis*, "Two Predictor Variables", Chapter 4, John Wiley & Sons, New York, 1981, pp. 193-212.

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Corhart Refractories Corporation's product brochure entitled "ZS-835 HD: Low Blistering Dense Zircon Refractory," 1995.

* cited by examiner

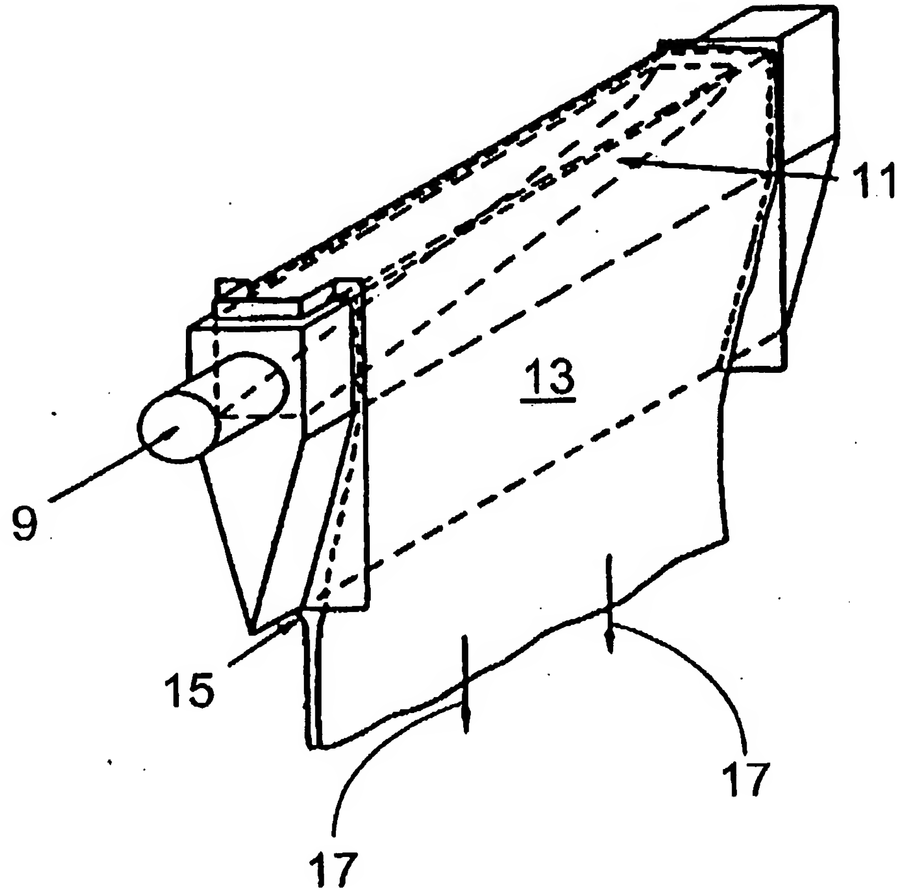


FIG. 1

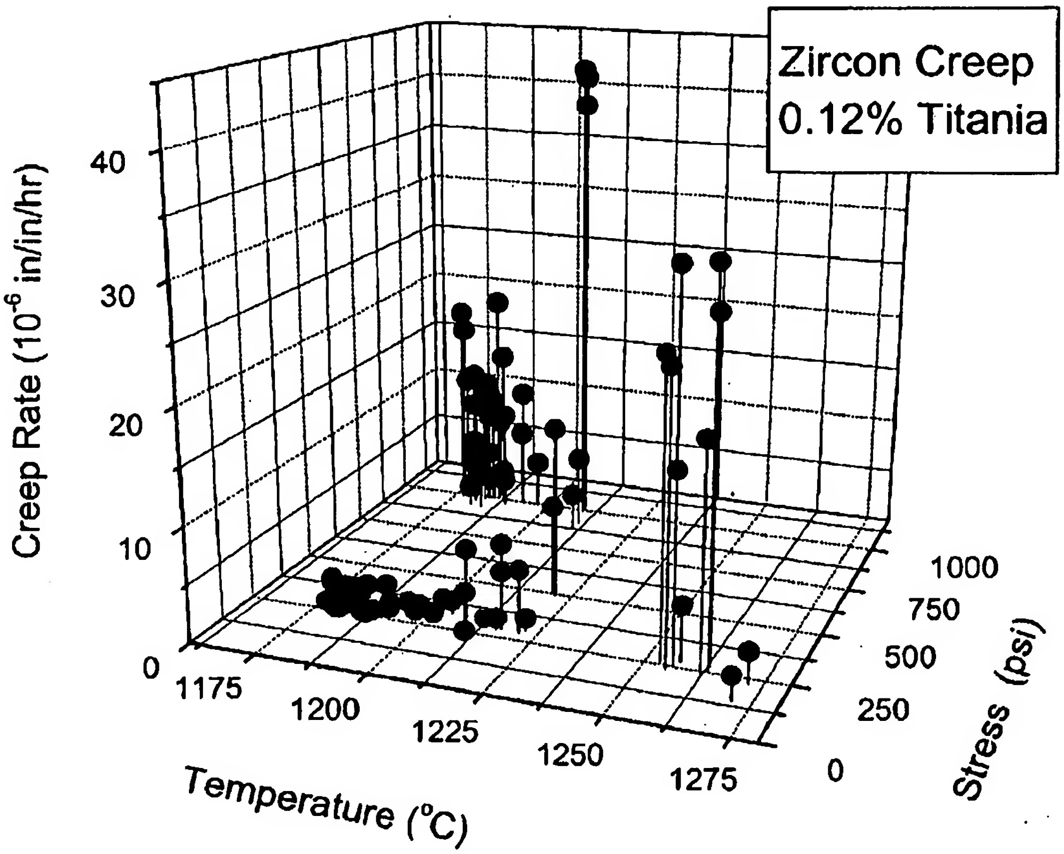
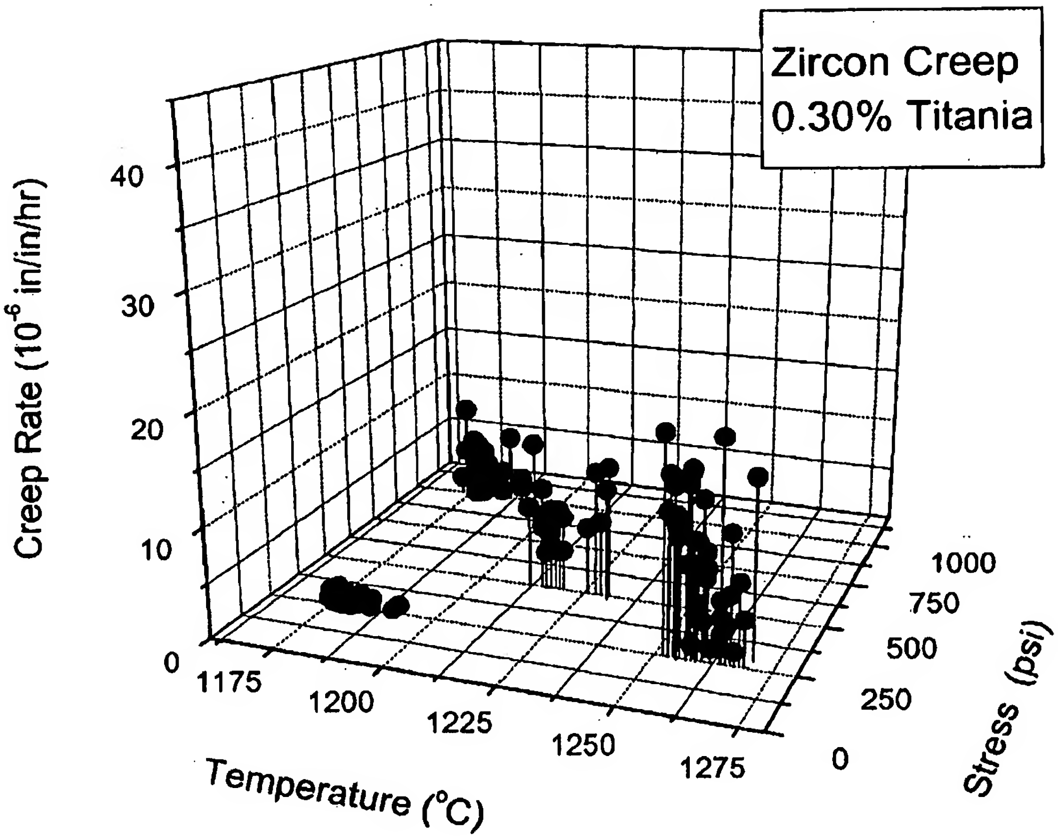

Zircon Creep
0.12% Titania
Creep Rate (10^{-6} in/in/hr)
40
30
20
10
0
1175
1200
1225
1250
1275
Temperature (°C)
1000
750
500
250
0
Stress (psi)

FIG. 2A



Zircon Creep
0.30% Titania
Creep Rate (10^{-6} in/in/hr)
40
30
20
10
0
1175
1200
1225
1250
1275
Temperature (°C)
1000
750
500
250
0
Stress (psi)

FIG. 2B

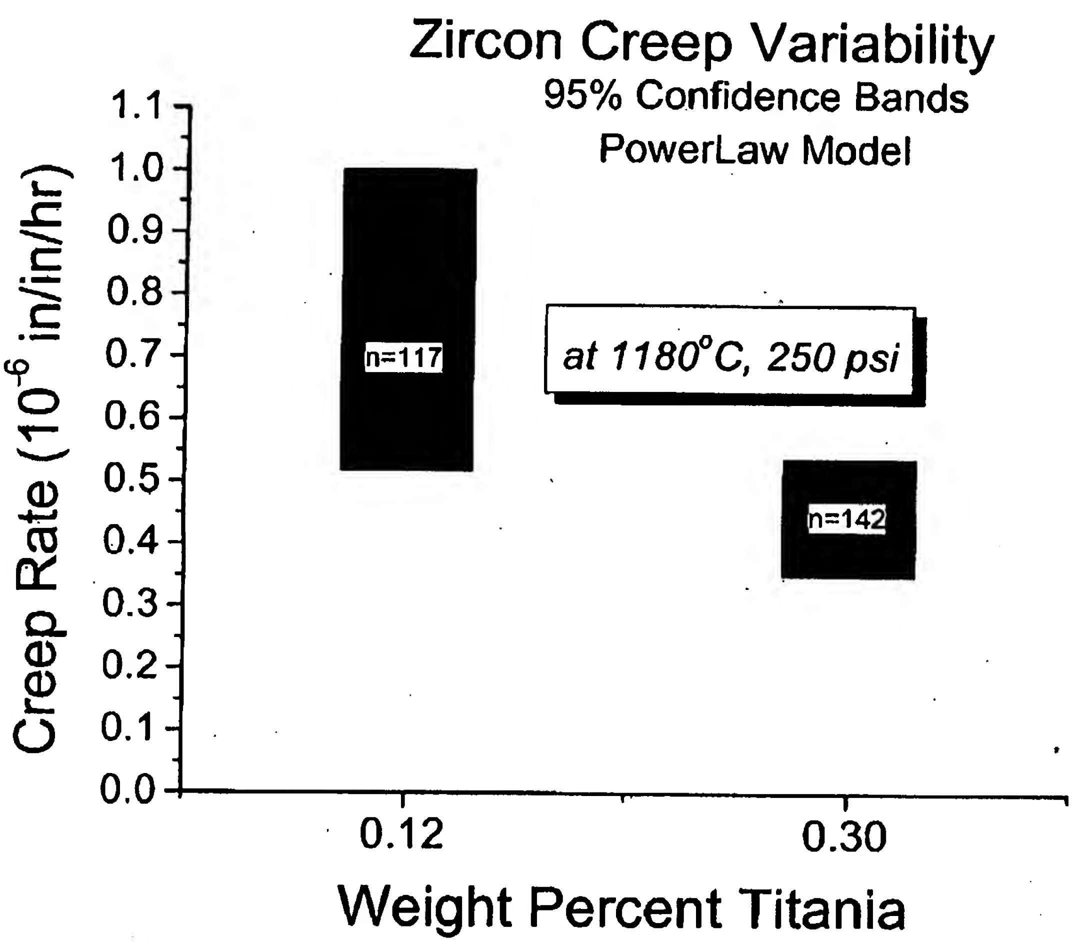
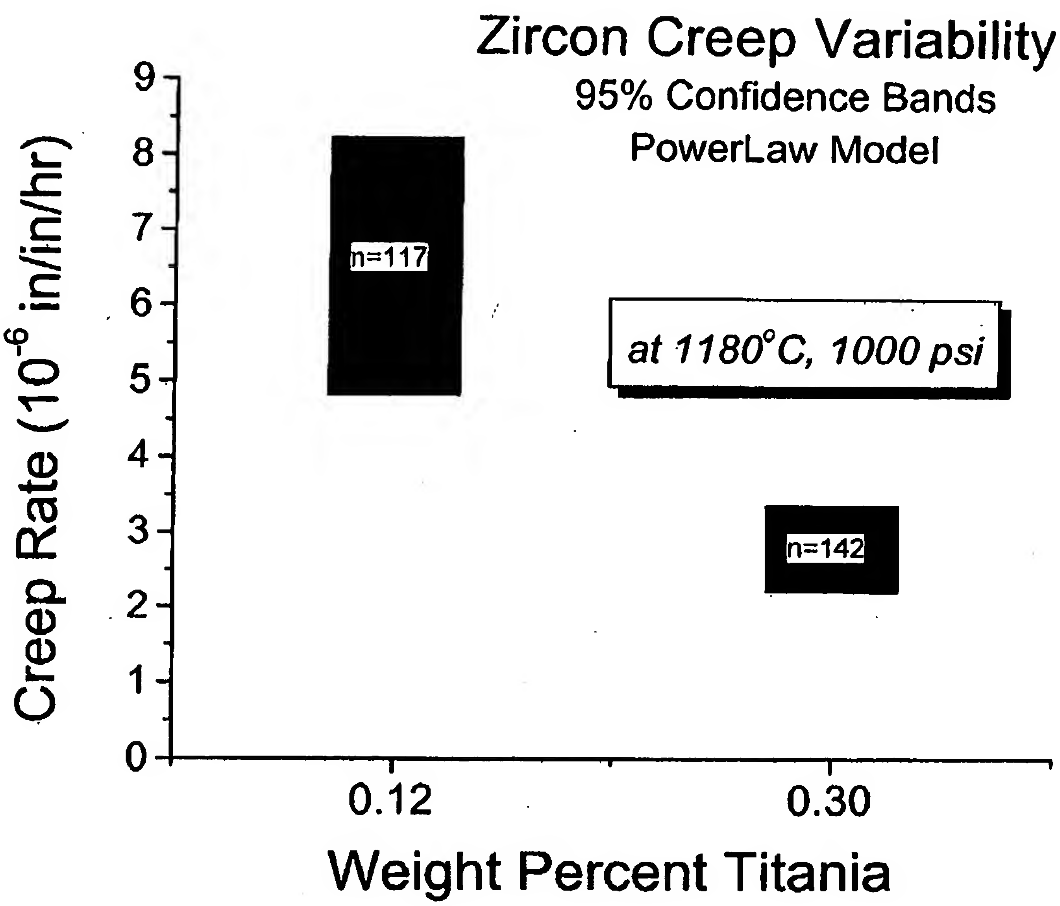

Zircon Creep Variability
95% Confidence Bands
PowerLaw Model
Creep Rate (10^{-6} in/in/hr)
1.1
1.0
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
n=117
at 1180°C, 250 psi
n=142
0.12
0.30
Weight Percent Titania

FIG. 3A



Zircon Creep Variability
95% Confidence Bands
PowerLaw Model
at 1180°C, 1000 psi
n=117
n=142
Creep Rate (10^-6 in/in/hr)
0
1
2
3
4
5
6
7
8
9
0.12
0.30
Weight Percent Titania

FIG. 3B

SAG CONTROL OF ISOPIPES USED IN MAKING SHEET GLASS BY THE FUSION PROCESS

CROSS REFERENCE TO RELATED APPLICATIONS

This is a continuation of co-pending International Application No. PCT/US01/45300, filed on Nov. 30, 2001, which was published in English under PCT Article 21(2) on Jun. 6, 2002 as International Publication No. WO 02/44102. This application claims the benefit under 35 USC §119(e) of U.S. Provisional Application No. 60/250,921, filed on Dec. 1, 2000.

NAMES OF PARTIES TO A JOINT RESEARCH AGREEMENT (35 USC §103(c)(2)(C))

This application is the result of a joint research agreement between Corning Incorporated and Corhart Refractories Corporation (now a part of Saint-Gobain Plastics and Ceramics, Inc.). The information specified in 37 CFR §1.71 (g)(1)(i)&(ii) is recorded in the assignment records of the U.S. Patent and Trademark Office at Reel 016156, Frame 0548.

FIELD OF THE INVENTION

This invention relates to isopipes used in the production of sheet glass by the fusion process and, in particular, to techniques for controlling the sag which such isopipes exhibit during use.

BACKGROUND OF THE INVENTION

A. The Fusion Process

The fusion process is one of the basic techniques used in the glass making art to produce sheet glass. See, for example, Varshneya, Arun K., "Flat Glass," *Fundamentals of Inorganic Glasses*, Academic Press, Inc., Boston, 1994, Chapter 20, Section 4.2., 534–540. Compared to other processes known in the art, e.g., the float and slot draw processes, the fusion process produces glass sheets whose surfaces have superior flatness and smoothness. As a result, the fusion process has become of particular importance in the production of the glass substrates used in the manufacture of liquid crystal displays (LCDs).

The fusion process, specifically, the overflow downdraw fusion process, is the subject of commonly assigned U.S. Pat. Nos. 3,338,696 and 3,682,609, to Stuart M. Dockerty, the contents of which are incorporated herein by reference. A schematic drawing of the process of these patents is shown In FIG. 1. As illustrated therein, the system includes a supply pipe 9 which provides molten glass to a collection trough 11 formed in a refractory body 13 known as an "isopipe."

Once steady state operation has been achieved, molten glass passes from the supply pipe to the trough and then overflows the top of the trough on both sides, thus forming two sheets of glass that flow downward and then inward along the outer surfaces of the isopipe. The two sheets meet at the bottom or root 15 of the isopipe, where they fuse together into a single sheet. The single sheet is then fed to drawing equipment (represented schematically by arrows 17), which controls the thickness of the sheet by the rate at which the sheet is drawn away from the root. The drawing equipment is located well downstream of the root so that the single sheet has cooled and become rigid before coming into contact with the equipment.

As can be seen in FIG. 1, the outer surfaces of the final glass sheet do not contact any part of the outside surface of the isopipe during any part of the process. Rather, these surfaces only see the ambient atmosphere. The inner surfaces of the two half sheets which form the final sheet do contact the isopipe, but those inner surfaces fuse together at the root of the isopipe and are thus buried in the body of the final sheet. In this way, the superior properties of the outer surfaces of the final sheet are achieved.

As is evident from the foregoing, isopipe 13 is critical to the success of the fusion process. In particular, the dimensional stability of the isopipe is of great importance since changes in isopipe geometry affect the overall success of the process. See, for example, Overman, U.S. Pat. No. 3,437,470, and Japanese Patent Publication No. 11-246230.

Significantly, the conditions under which the isopipe is used make it susceptible to dimensional changes. Thus, the isopipe must operate at elevated temperatures on the order of 1000° C. and above. Moreover, in the case of the overflow downdraw fusion process, the isopipe must operate at these elevated temperatures while supporting its own weight as well as the weight of the molten glass overflowing its sides and in trough 11, and at least some tensional force that is transferred back to the isopipe through the fused glass as it is being drawn. Depending on the width of the glass sheets that are to be produced, the isopipe can have an unsupported length of 1.5 meters or more.

To withstand these demanding conditions, isopipes 13 have been manufactured from isostatically pressed blocks of refractory material (hence the name "iso-pipe"). In particular, isostatically pressed zircon refractories have been used to form isopipes for the fusion process. As known in the art, zircon refractories are materials composed primarily of ZrO_2 and SiO_2, e.g., in such materials, ZrO_2 and SiO_2 together comprise at least 95 wt. % of the material, with the theoretical composition of the material being $ZrO_2.SiO_2$ or, equivalently, $ZrSiO_4$. Even with such high performance materials, in practice, isopipes exhibit dimensional changes which limit their useful life. In particular, isopipes exhibit sag such that the middle of the unsupported length of the pipe drops below its outer supported ends. The present invention is concerned with controlling such sag.

A primary contributor to the sag of an isopipe is the creep rate $\dot{\epsilon}=d\epsilon/dt$ of the material from which it is made. As known in the art, for many materials, creep rate as a function of applied stress σ can be modeled by a power law expression of the following form:

$$\dot{\epsilon}=A\sigma^n\exp(Q/T) \quad (1)$$

where T is temperature and A, n, and Q are material dependent constants. See Kingery et al., "Plastic Deformation, Viscous Flow, and Creep," *Introduction to Ceramics*, 2nd edition, John Wiley & Sons, New York, 1976, 704–767 and, in particular, equation 14.9. Being the time derivative of strain, the units of creep rate are length/length/time. Because in equation (1) creep rate varies as stress raised to a power, i.e., σ^n, the use of equation (1) will be referred to herein as the "power law model."

Lowering the creep rate of the material used to make an isopipe results in less sag during use. As discussed in detail below, in accordance with certain aspects of the invention it has been found that the sag of an isopipe can be reduced by forming the isopipe from an isostatically pressed zircon refractory having a titania (TiO_2) content which is greater than 0.2 wt. % and less than 0.4 wt. %, e.g., a TiO_2 content of approximately 0.3 wt. %. In particular, it has been found

that such a zircon refractory exhibits a lower mean creep rate than zircon refractories used in the past to from isopipes and having a titania content of about 0.1 wt. %.

In addition, it has also been found that controlling the titania content of a zircon refractory to be within the above range significantly enhances the usefulness of the power law model of equation (1) in modeling the sag of isopipes during use. This enhanced usefulness results from improved 95% confidence intervals for the mean creep rates predicted by the model when equation (1) is evaluated for a particular set of σ,T values. Such improved 95% confidence intervals, in turn, mean that the sag which an isopipe will exhibit during use can be more accurately modeled using, for example, a finite element or other modeling technique. More accurate modeling greatly enhances the ability to develop improved isopipe designs since numerous designs can be evaluated theoretically with only the best candidates being selected for actual construction and testing.

B. Zircon Refractories

As indicated above, the present invention relates to isopipes composed of a zircon refractory having a titania concentration within specified limits. Corhart Refractories Corporation (Louisville, Ky.) offers a number of zircon refractories containing varying amounts of TiO_2. For example, Corhart's ZS-835 product is specified to contain 0.2 wt. % TiO_2, its ZS-835HD product 0.4 wt. %, its Zircon 20 product 0.7 wt. %, and its ZS-1300 product 1.2 wt. %.

As a raw material, zircon can have varying amounts of titania. For example, U.S. Pat. No. 2,752,259 reports that the zircon used in its examples had 0.34 wt. % TiO_2, while the zircon used in U.S. Pat. No. 3,285,757 had 0.29 wt. % TiO_2. U.S. Pat. Nos. 3,347,687 and 3,359,124 each describe zircons having TiO_2 concentrations of 0.2 wt. %. In addition to being naturally present in zircon as a raw material, TiO_2 can also be a component of clays used in producing zircon refractories. See U.S. Pat. Nos. 2,746,874 and 3,359,124.

Other discussions of the use of titania in zircon products can be found in Goerenz et al., U.S. Pat. No. 5,407,873 which discloses (1) the use of phosphorus compounds to improve the corrosion resistance of zirconium silicate bricks and (2) the use of titanium dioxide as a sintering aid in the manufacture of such bricks. Although the patent states that sintering can be improved by adding between 0.1 wt. % and 5 wt. % of titanium dioxide, all of the examples of the patent use more than 1 wt. % of titanium dioxide and the patent's preferred composition consists of 98 wt. % zirconium silicate, 1.5 wt. % titanium dioxide, and 0.5 wt. % of a phosphorous compound.

Wehrenberg et al., U.S. Pat. No. 5,124,287 relates to the use of zirconia in particle form to improve the thermal shock resistance of zircon refractories. Titania is employed to enhance grain growth during sintering. The patent claims titania concentrations between 0.1 wt. % and 4 wt. %. The preferred titania concentration is 1 wt. %, and when blistering is a problem, only 0.1 wt. % titania is used. The patent states that "grog" having a titania concentration of 0.2 wt. % was used as a starting material for some of its examples.

Significantly, none of the foregoing disclosures regarding the use of titania in zircons relates to employing titania concentration as a means to control the creep rate of a zircon refractory, or to enhance the ability of a power law model to represent the material, or to achieve the ultimate goal of reducing the sag of an isopipe made of a zircon refractory.

SUMMARY OF THE INVENTION

In view of the foregoing, it is an object of this invention to provide improved isopipes for use in the fusion process. More particularly, it is an object of the invention to provide isopipes that exhibit less sag than existing isopipes.

To achieve the foregoing and other objects, the invention in accordance with a first aspect provides isopipes which comprise a zircon refractory that exhibits a lower creep rate than the zircon refractories previously used to produce isopipes.

In accordance with a second aspect, the invention provides isopipes which comprise a zircon refractory that in comparison to zircon refractories previously used to produce isopipes, has a creep rate that can be modeled more accurately by a power law model.

In accordance with a third aspect, the invention provides isopipes comprising a body having a configuration adapted for use in a fusion process, said body comprising a zircon refractory which purposely comprises TiO_2 at a concentration greater than 0.2 wt. % and less than 0.4 wt. %, preferably greater than 0.25 wt. % and less than 0.35 wt. %, and most preferably about 0.3 wt. %.

In accordance with a fourth aspect, the invention provides isopipes comprising a body having a configuration adapted for use in a fusion process, said body comprising a zircon refractory which has a mean creep rate (MCR) at 1180° C. and 250 psi of less than 0.7×10^{-6} inches/inches/hour, preferably less than 0.6×10^{-6} inches/inches/hour, and most preferably less than 0.5×10^{-6} inches/inches/hour, where the MCR is determined using a power law model, i.e., a power law model fit to experimental data.

In accordance with this fourth aspect, the zircon refractory also preferably has a MCR at 1180° C. and 1000 psi of less than 5×10^{-6} inches/inches/hour and more preferably less than 3×10^{-6} inches/inches/hour, where again the MCR is determined using a power law model.

In accordance with a fifth aspect, the invention provides isopipes comprising a body having a configuration adapted for use in a fusion process, said body comprising a zircon refractory which has a MCR at 1180° C. and 250 psi and a 95 percent confidence band (CB) for said MCR such that the CB to MCR ratio is less than 0.5, the MCR and the CB both being determined using a power law model. In accordance with these aspects of the invention, the CB to MCR ratio at 1180° C. and 1000 psi is also preferably less than 0.5, where again the MCR and the CB values used to calculate the CB to MCR ratio at said temperature and stress level are determined using a power law model.

In accordance with a sixth aspect, the invention provides a method for reducing the sag of an isopipe used in a fusion process that produces glass sheets comprising forming said isopipe from a zircon refractory which purposely comprises TiO_2 at a concentration greater than 0.2 wt. % and less than 0.4 wt. %, preferably greater than 0.25 wt. % and less than 0.35 wt. %, and most preferably about 0.3 wt. %.

The above first through sixth aspects of the invention can be used separately or in all possible combinations. For example, the compositional limitations of the third and sixth aspects of the invention (including the base, preferred, and most preferred values of those limitations) can be combined with the mean creep rate limitations of the fourth aspect of the invention (including the base, preferred, and most preferred values of those limitations) and/or with the CB to MCR ratio limitations of the fifth aspect of the invention (including the base and preferred pressure values of those limitations). Similarly, the mean creep rate limitations of the

fourth aspect of the invention (including the base, preferred, and most preferred values of those limitations) can be combined with CB to MCR ratio limitations of the fifth aspect of the invention (including the base and preferred pressure values of those limitations).

As used in this specification and in the claims, the term "isopipe" means any sheet forming delivery system used in a fusion process which produces flat glass wherein at least a part of the delivery system comes into contact with the glass just prior to fusion, irrespective of the configuration or the number of components making up the delivery system. Also, the MCR and CB values are determined using standard statistical techniques for calculating such values from the fit of an equation such as the power law model to measured data. See, for example, Draper et al., *Applied Regression Analysis*, John Wiley & Sons, New York, 1981, 193–212.

Further, the word "purposely" when used in connection with TiO_2 concentrations means that the TiO_2 concentration is intentionally selected to control isopipe sag and is not merely a TiO_2 concentration which one or more zircons (including zircons for making isopipes) may have had as a result of compositional variations without being the result of a conscious intention to control isopipe sag and/or to improve the ability of a power law model to represent the creep rate of zircon used in an isopipe.

Additional features and advantages of the invention will be set forth in the detailed description which follows, and in part will be readily apparent to those skilled in the art from that description or recognized by practicing the invention as described herein. It is to be understood that both the foregoing general description and the following detailed description are merely exemplary of the invention, and are intended to provide an overview or framework for understanding the nature and character of the invention as it is claimed. The accompanying drawings are included to provide a further understanding of the invention, and are incorporated in and constitute a part of this specification. The drawings illustrate various aspects of the invention, and together with the description serve to explain the principles and operation of the invention.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a schematic drawing illustrating a representative construction for an isopipe for use in an overflow downdraw fusion process for making flat glass sheets.

FIGS. 2A and 2B are three dimensional plots showing experimentally measured creep rate as a function of temperature and stress for zircon specimens having TiO_2 concentrations of 0.12 wt. % and 0.30 wt. %, respectively.

FIGS. 3A and 3B are plots illustrating the differences in creep rate variability at a temperature of 1180° C. for 0.12 wt. % TiO_2 versus 0.30 wt. % TiO_2 for applied stresses of 250 psi and 1000 psi, respectively.

DETAILED DESCRIPTION OF THE INVENTION

As discussed above, the present invention relates to the use of zircon refractories to produce isopipes for use in a fusion process where the zircon refractory has a TiO_2 content greater than 0.2 wt. % and less than 0.4 wt. %.

Such a TiO_2 content causes the isopipe to exhibit reduced sag as a result of the refractory having a lower mean creep rate than zircon refractories currently used in the art. For example, the zircon refractory can have a mean creep rate at 1180° C. and 250 psi substantially less than 0.5×10^{-6} inches/inches/hour.

In addition, such a TiO_2 content also causes the refractory to have a 95% confidence band (CB) for said mean creep rate (MCR) which is less than 50% of the mean creep rate, i.e., CB/MCR<0.5. Such a confidence band reduces the chances that the zircon refractory of a particular isopipe will have an abnormally high creep rate and thus cause the isopipe to have a short lifetime as a result of exhibiting unacceptable sag prematurely.

The TiO_2 content of a zircon refractory can be determined using various techniques known in the art. For example, the content can be determined by means of an X-ray fluorescence analysis (XRF). The titania content of the refractory can be adjusted so that the final product has the desired TiO_2 content by incorporating TiO_2 as needed in the batch materials used to prepare the refractory. Thereafter, the refractory can be prepared in accordance with techniques currently known in the art or with improved techniques which may be developed in the future.

Similarly, isopipes can be prepared from the zircon refractories of the invention using techniques currently known in the art or with improved techniques which may be developed in the future. Typically, the isopipe will be prepared by being machined from a single block of the zircon refractory, although other approaches can be used if desired.

Without intending to limit it in any manner, the present invention will be more fully described by the following examples.

Lots of zircon refractories containing 0.12 wt. % or 0.30 wt. % TiO_2 were obtained from Corhart Refractories Corporation (Louisville, Ky.). Each lot represented a separate firing and typically included multiple blocks of material of suitable dimensions to produce an isopipe, i.e., the blocks had lengths greater than 1.5 meters.

Creep rate tests were performed on 117 specimens taken from blocks having 0.12 wt. % TiO_2 and 142 specimens taken from blocks having 0.30 wt. % TiO_2. A three point flexure technique was used to determine creep rates in which a bar of the material being tested was supported at its ends and loaded at its center. The applied stress in pounds per square inch (psi) was determined in accordance with conventional procedures as set forth in ASTM C-158. In particular, applied stress σ was determined from the relation:

$$\sigma=3\cdot AL\cdot SS/(2\cdot SW\cdot SH^2)$$

where AL=applied load, SS=support span, SW=specimen width, and SH=specimen height.

The bar was heated and its flexure as a function of time was measured. A midspan deflection rate was obtained by calculating the slope of the resulting deflection versus time plot once steady state conditions for the particular load and temperature had been reached. In particular, the midspan deflection rate was determined for the "secondary creep" portion of the strain versus time curve. See, for example, the Kingery et al. text cited above at pages 707–709.

Creep rates $\dot{\epsilon}$ were then obtained from the relation:

$$\dot{\epsilon}=DR\cdot 2\cdot SH/SS^2$$

where SH and SS are as defined above and DR=midspan deflection rate.

FIG. 2A and FIG. 2B are three dimensional plots of the creep rate values obtained in this way for the 0.12 wt. % TiO_2 and 0.30 wt. % TiO_2 specimens, respectively. The reduction in the scatter of the data achieved by the change in TiO_2 concentration is immediately evident from these figures. In terms of producing isopipes which will have repeatable creep properties, the data of these figures show

that a zircon refractory having a TiO_2 content of around 0.3 wt. % is clearly much better than one having a TiO_2 content around 0.1 wt. %.

The power law model of equation (1) was fit to the data of FIG. 2A and to that of FIG. 2B using a commercial data analysis package, namely, "TableCurve 3D: Automated Surface Fitting and Equation Discovery," Version 3.0 for Windows® 95 & NT, software and documentation, SPSS Inc., Chicago, 1997 (hereinafter the "TABLE CURVE 3D program"). The values of the material dependent constants A, n, and Q obtained in this way for the two cases are set forth in Table 1.

Using these constants and the TABLE CURVE 3D program, mean creep rates and 95% confidence bands were determined for a temperature of 1180° C. and a stress of 250 psi, which are representative of the temperatures and stress levels which an isopipe will typically experience during use. The results of this analysis are shown in FIG. 3A.

Two important facts are evident from this figure. First, the mean creep rate has been substantially reduced as a result of the increase in TiO_2 content from 0.12 wt. % to 0.30 wt. %. This means that isopipes composed of zircon refractories having higher TiO_2 than previously used will exhibit less sag during use, a highly desirable result. Moreover, the size of the 95% confidence band has also been substantially reduced by the increase in TiO_2 content. This means that an individual isopipe made from an individual block of a zircon refractory is more likely to have its creep rate closer to the predicted mean creep rate when the TiO_2 content of the material is increased than when it is not increased, another highly desirable result since predictability in a manufacturing setting makes for more efficient planning and operation.

To further demonstrate the controlling effect which TiO_2 content has on creep rate, mean creep rates and 95% confidence bands were also determined for a stress of 1000 psi, again using the constants of Table 1 and the TABLE CURVE 3D program. The results are shown in FIG. 3B. The reduction in mean creep rate achieved by increasing the TiO_2 content is even greater at this higher stress level.

Table 2 summarizes the results of using the TABLE CURVE 3D program to determine mean creep rates and 95% confidence bands for the data of FIG. 2. Zircon refractories having a TiO_2 content above and below the 0.3 wt. % value used to generate this data will exhibit similar MCR and CB values to those shown in Table 2. In particular, reduced MCR and CB/MCR values compared to previously used zircon refractories are achieved when the TiO_2 content of the refractory is greater than 0.2 wt. %. The improved performance continues as the TiO_2 content is increased above 0.3 wt. %. However, oxygen blisters can be generated at the isopipe/glass interface when the TiO_2 content of the zircon refractory reaches about 0.4 wt. %. Thus, in accordance with the invention, the TiO_2 content of the refractory should be above 0.2 wt. % but below 0.4 wt. %.

Although specific embodiments of the invention have been discussed, a variety of modifications to those embodiments which do not depart from the scope and spirit of the invention will be evident to persons of ordinary skill in the art from the disclosure herein. The following claims are intended to cover the specific embodiments set forth herein as well as such modifications, variations, and equivalents.

TABLE 1

TiO_2 (wt. %)	A	n	Q
0.12.	1.04×10^{12}	1.56	-73302
0.30	1.20×10^{14}	1.33	-79038

TABLE 2

Example	TiO_2 (wt. %)	T (° C.)	σ (psi)	MCR (10^{-6} in/in/hr)	CB (10^{-6} in/in/hr)	CB/MCR
1	0.12	1180	250	0.7197	0.5163 to 1.003	0.6763
2	0.30	1180	250	0.4340	0.3500 to 0.5390	0.4355
3	0.12	1180	1000	6.296	4.811 to 8.240	0.5446
4	0.30	1180	1000	2.730	2.210 to 3.380	0.4286

MCR = mean creep rate
CB = 95% confidence band for the MCR

What is claimed is:

1. An isopipe comprising a body having a configuration adapted for use in a fusion process, said body comprising a zircon refractory which (i) comprises TiO_2 at a concentration greater than 0.2 wt. % and less than 0.4 wt. % and (ii) has a mean creep rate at 1180° C. and 250 psi of less than 0.7×10^{-6} inches/inches/hour, where the mean creep rate is determined using a power law model.

2. The isopipe of claim 1 wherein the zircon refractory comprises TiO_2 at a concentration greater than 0.25 wt. % and less than 0.35 wt. %.

3. The isopipe of claim 1 wherein the zircon refractory comprises TiO_2 at a concentration of about 0.3 wt. %.

4. The isopipe of claim 1 wherein the mean creep rate is less than 0.5×10^{-6} inches/inches/hour.

5. The isopipe of claim 1 wherein the zircon refractory has a mean creep rate at 1180° C. and 1000 psi of less than 3.0×10^{-6} inches/inches/hour, where the mean creep rate is determined using a power law model.

6. The isopipe of claim 1 wherein the mean creep rate (MCR) at 1180° C. and 250 psi has a 95 percent confidence band (CB) such that the CB to MCR ratio is less than 0.5, said 95 percent confidence band being determined using a power law model.

7. The isopipe of claim 1 wherein the zircon refractory has a mean creep rate (MCR) at 1180° C. and 1000 psi and a 95 percent confidence band (CB) for said mean creep rate such that the CB to MCR ratio is less than 0.5, said mean creep rate and said 95 percent confidence band being determined using a power law model.

8. An isopipe comprising a body having a configuration adapted for use in a fusion process, said body comprising a zircon refractory which: (i) comprises TiO_2 at a concentration greater than 0.2 wt. % and (ii) has a mean creep rate at 1180° C. and 250 psi of less than 0.7×10^{-6} inches/inches/hour, where the mean creep rate is determined using a power law model.

9. The isopipe of claim 8 wherein the mean creep rate is less than 0.6×10^{-6} inches/inches/hour.

10. The isopipe of claim 8 wherein the mean creep rate is less than 0.5×10^{-6} inches/inches/hour.

11. The isopipe of claim 8 wherein the mean creep rate (MCR) at 1180° C. and 250 psi has a 95 percent confidence band (CB) such that the CB to MCR ratio is less than 0.5, said 95 percent confidence band being determined by a power law model.

12. The isopipe of claim 8 wherein the zircon refractory has a mean creep rate at 1180° C. and 1000 psi of less than 5×10^{-6} inches/inches/hour, where the mean creep rate is determined using a power law model.

13. The isopipe of claim 12 wherein the mean creep rate at 1180° C. and 1000 psi is less than 3×10^{-6} inches/inches/hour.

14. The isopipe of claim 12 wherein the mean creep rate (MCR) at 1180° C. and 1000 psi has a 95 percent confidence band (CB) such that the CB to MCR ratio is less than 0.5, said 95 percent confidence band being determined by a power law model.

15. An isopipe comprising a body having a configuration adapted for use in a fusion process, said body comprising a zircon refractory which: (i) comprises TiO_2 at a concentration greater than 0.2 wt. % and (ii) has a mean creep rate (MCR) at 1180° C. and 250 psi and a 95 percent confidence band (CB) for said mean creep rate such that the CB to MCR ratio is less than 0.5, the MCR and the GB both being determined using a power law model.

16. The isopipe of claim 15 wherein the zircon refractory has a mean creep rate (MCR) at 1180° C. and 1000 psi and a 95 percent confidence band (CB) for said mean creep rate such that the CB to MCR ratio is less than 0.5, the MCR and the CB both being determined using a power law model.

17. A method for reducing the sag of an isopipe used in a fusion process that produces glass sheets comprising forming said isopipe from a zircon refractory which (i) comprises TiO_2 at a concentration greater than 0.2 wt. % and less than 0.4 wt. % and (ii) has a mean creep rate at 1180° C. and 250 psi of less than 0.7×10^{-6} inches/inches/hour, where the mean creep rate is determined using a power law model.

18. The method of claim 17 wherein the zircon refractory comprises TiO_2 at a concentration greater than 0.25 wt. % and less than 0.35 wt. %.

19. The method of claim 17 wherein the zircon refractory comprises TiO_2 at a concentration of about 0.3 wt %.

20. The method of claim 17 wherein the mean creep rate is less than 0.5×10^{-6} inches/inches/hour.

21. The method of claim 17 wherein the zircon refractory has a mean creep rate at 1180° C. and 1000 psi of less than 3.0×10^{-6} inches/inches/hour, where the mean creep rate is determined using a power law model.

22. The method of claim 17 wherein the mean creep rate (MCR) at 1180° C. and 250 psi has a 95 percent confidence band (CB) such that the CB to MCR ratio is less than 0.5, said 95 percent confidence band being determined using a power law model.

23. The method of claim 17 wherein the zircon refractory has a mean creep rate (MCR) at 1180° C. and 1000 psi and a 95 percent confidence band (CB) for said mean creep rate such that the CB to MCR ratio is less than 0.5, said mean creep rate and said 95 percent confidence band being determined using a power law model.

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